

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
28 June 2001 (28.06.2001)

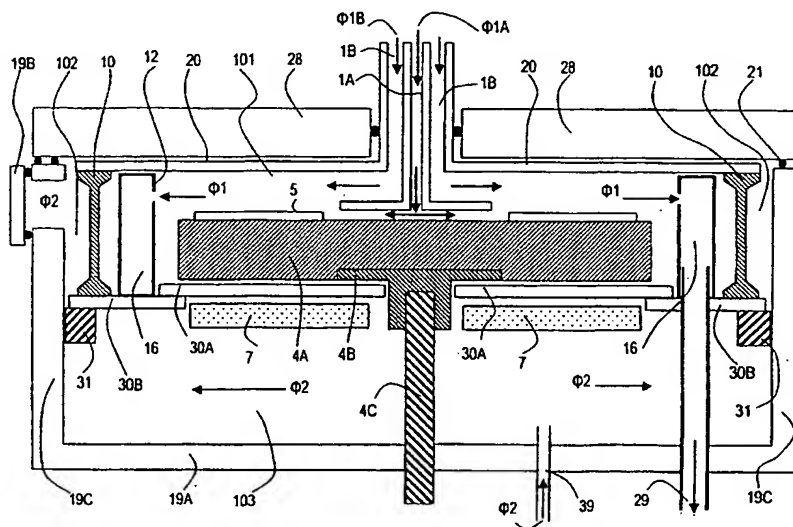
PCT

(10) International Publication Number
WO 01/46498 A3

- (51) International Patent Classification⁷: C30B 25/14, (74) Agents: GRUNDMANN, Dirk et al.; Corneliusstrasse 45, C23C 16/44 42329 Wuppertal (DE).
- (21) International Application Number: PCT/EP00/11992 (81) Designated States (*national*): JP, KR, US.
- (22) International Filing Date: 30 November 2000 (30.11.2000) (84) Designated States (*regional*): European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR).
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data: 99403255.5 22 December 1999 (22.12.1999) EP
- (88) Date of publication of the international search report: 23 May 2002
- (71) Applicant (*for all designated States except US*): AIX-TRON AG [DE/DE]; Kackertstrasse 15-17, 52072 Aachen (DE).
- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

- (72) Inventor; and
- (75) Inventor/Applicant (*for US only*): FRIJLINK, Peter [NL/FR]; 25, rue Frédéric Mistral, F-91330 Yerres (FR).

(54) Title: CHEMICAL VAPOR DEPOSITION REACTOR AND PROCESS CHAMBER FOR SAID REACTOR



(57) Abstract: A chemical vapor deposition reactor having a process chamber (101) accommodating a substrate holder (4a) for wafers (5), a first gas flow ($\Phi 1$) of reactive gases to process the wafers and a crown-shaped gas-collector surrounding the substrate-holder, wherein said reactor further comprises: a base plate (30) and a cover plate (20) disposed respectively beneath and above the substrate-holder, an outer ring (10) surrounding the gas-collector (16) and touching both the base plate (30) and the cover-plate (20), and a second flow ($\Phi 2$) of non-reactive gases propagating in spaces outside the process chamber limited by the base and cover plates and the outer ring, and said second flow acting as a counter-flow ($\Phi 2$) for preventing the first reactive gas flow ($\Phi 1$) to exit from the process chamber but through the gas-collector (16). Application: Chemical vapor Deposition Reactors for MOVPE Deposition Methods.

BEST AVAILABLE COPY

WO 01/46498 A3

INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 00/11992

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C30B25/14 C23C16/44

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C30B C23C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0 366 173 A (PHILIPS ELECTRONIQUE LAB ;PHILIPS NV (NL)) 2 May 1990 (1990-05-02) claim 1 ---	1
A	US 4 450 786 A (DOEHLER JOACHIM ET AL) 29 May 1984 (1984-05-29) claims 1-4 ---	1,2
A	WO 99 42636 A (JUERGENSEN HOLGER ;SCHUMACHER MARKUS (DE); DESCHLER MARC (DE); AIX) 26 August 1999 (1999-08-26) claim 1 ---	1
A	US 4 961 399 A (FRIJLINK PETER M) 9 October 1990 (1990-10-09) cited in the application -----	

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents:

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- *B* document member of the same patent family

Date of the actual completion of the international search

7 June 2001

Date of mailing of the international search report

15/06/2001

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel (431-70) 340-2040 Telex 31 651 epo nl

Authorized officer

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 00/11992

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0366173 A	02-05-1990	FR 2638020 A	20-04-1990
		DE 68908335 D	16-09-1993
		DE 68908335 T	24-02-1994
		JP 1871976 C	26-09-1994
		JP 2157189 A	15-06-1990
		JP 5085520 B	07-12-1993
		US 4976217 A	11-12-1990
US 4450786 A	29-05-1984	US 4438724 A	27-03-1984
		AT 18680 T	15-04-1986
		BR 8304258 A	24-04-1984
		CA 1194617 A	01-10-1985
		DE 3362604 D	24-04-1986
		EP 0101286 A	22-02-1984
		ES 524944 D	16-11-1984
		ES 8501568 A	16-02-1985
		MX 159116 A	21-04-1989
		PH 19757 A	26-06-1986
		AU 556804 B	20-11-1986
		AU 1764683 A	16-02-1984
		JP 1705829 C	27-10-1992
		JP 3067333 B	22-10-1991
		JP 59063723 A	11-04-1984
WO 9942636 A	26-08-1999	DE 19813523 A	07-10-1999
		DE 19980266 D	25-05-2000
		EP 0975821 A	02-02-2000
US 4961399 A	09-10-1990	FR 2628984 A	29-09-1989
		DE 68908927 D	14-10-1993
		DE 68908927 T	24-03-1994
		EP 0334433 A	27-09-1989
		JP 1278497 A	08-11-1989
		JP 2835338 B	14-12-1998
		KR 137876 B	17-08-1998

This base plate 30 is arranged within the reactor in such a way that the reactive gases are in contact with the wafers. The reactor comprises a cylindrical body 19C having a vertical axis, which surrounds the base plate 30. On the lower and upper parts of said cylindrical body 19C are placed a bottom plate 19A and a roughly flat cover 28, respectively.

5 The cylindrical body 19C being in a cold area of the reactor, the tightness with the bottom plate 19A and the cover 28 is ensured by toric joints 21, the bottom plate being preferably an horizontal part 19A of the cylindrical body 19C. The cover 28 can be lifted to place the base plate 30. It is practically parallel to the base plate during the process operation, in which reactive gases are introduced into the reactor through concentric funnels forming an

10 inlet 1A for a first precursor of reactive gas flow $\Phi 1A$ and an inlet 1B for a second precursor of reactive gas flow $\Phi 1B$. The gas flows move radially above the base plate 30 towards the periphery of said base plate. The reactive process gases are collected by a gas-collector 16 through outlets 12, and by a gas plenum 29 connected to the gas-collector 16 at an exhaust port 40. The gas-collector 16 is disposed on the edge of the base plate 30, which base plate

15 is positioned unto a ring 31.

Said gas-collector is described in detail in reference to FIG.5A. This gas-collector is crown-shaped and comprises an annular top part 15, and an annular low part 14, said top and low parts being connected by lateral flanges 17 and 18. Screws 13 regularly spaced hold the top part 15 and the low part 14 together with the flanges 17, 18. The processing

20 chamber 101 is limited by the flange 18 of the crown-shaped gas-collector 16. The flange 18 surrounding the substrate holder 4A has apertures 12 regularly spaced to let the reactive gases exhaust from the process chamber 101 and be lead by the gas-collector 16 towards a gas plenum 29. Referring to FIG.5B, the gas plenum 29 is a tube having a wall 29A, which is provided with external longitudinal ridges 29B. The gas plenum 29 slides into an aperture 40

25 of the annular low part 14, leaving small apertures 29C between the internal part 41 of the aperture 40 and the wall 29A of the gas plenum tube. The metal for forming the gas-collector may be favorably molybdenum. This gas-collector made of molybdenum presents the advantages of not involving the risk of exerting excessive forces onto the cover plate and base plate, of supporting the temperature prevailing in the chamber at the level of the

30 semiconductor wafers and of being resistant to the processing gases use. Preferably, two tubes 29 arranged diametrically opposite to each other merge into the interior of the gas-collector 16 to discharge the gases to the exterior.

In the example of embodiment illustrated by FIG.1, the inlet tube 1b has an extension 20 that is bound to the cover 28, so that the inlet tubes 1A, 1B are lifted together

35 with the cover 28 during an opening/closing operation of the reactor and so that this extension 20 forms the actual cover, hereafter referred to as cover plate 20, for the space immediately above the wafers 5 and substrate holder 4A. Preferably the base plate 30, cover

plate 20 and inlets 1A, 1B are made of quartz that is a non-porous material resilient to the high temperatures of the process.

According to the invention, isolation means prevent the reactive gases from flowing into spaces of the reactor other than the space immediately above the substrate holder and the wafers, referred to as first space 101, and hereafter denoted process chamber, in fact containing the substrate holder 4A with the wafers 5, the inlets 1A, 1B and the gas-collector 16. Said isolation means comprise in detail :

A cylindrical isolation element referred to as outer ring 10. Said outer ring 10 surrounds the gas-collector 16 and the substrate holder 4A with the wafers 5 and touches both the base plate 30 and the cover plate 20, in such a manner that the first space denoted process chamber 101 containing the process gases is isolated from a second space 102 and third space 103 disposed externally to the outer ring 10 and plates 30 and 20, within the peripheral wall 19C, 19A. The outer ring 10 is disposed on the edge of the base plate 30, and has upper and lower flat surfaces for touching the base plate 30 and the extension cover 20. This outer ring 10 is preferably made of inox.

It is to be noted that the gas-collector 16 is not necessarily part of the isolating elements. It stands on the edge of the base plate 30 in such a way that it does not touch the cover plate 20 for allowing dilations due the high temperature gases collected.

A non-porous annular element 4B bonded to the porous substrate holder 4A and to the rotating axis 4C. Said annular non-porous element 4B has an outer diameter that is greater than the diameter of the opening 32 for the rotating axis 4C, in order to prevent the reactive gases to exit through said opening 32. As a matter of fact, the base plate 30 is heated by the heating elements 7 at a temperature above 300°C, so the element 4B is constituted of a refractory metal apt to admit such temperatures. The non-porous element 4B of the substrate holder 4A is associated to the base plate 30 to particularly isolate the process chamber 101 from the third space 103.

A counter-flow $\Phi 2$ of non-reactive gas is applied in areas far from the reactive gases i. e. is introduced in the peripheral third space 103 through an inlet 39. This counter-flow propagates in the space 102 and is applied to the process chamber in a direction opposite to the reactive gases by way of specific structures referred to as high temperature seal means.

Said seal means are small grooves or roughened zones of surfaces of the interfaces of the isolation elements above-described. In the embodiment of FIG.1, the seal means structures are applied to the flat contact surfaces of the outer ring 10 at the interfaces with the cover plate 20 and the base plate 30. The seal means structures according to the invention avoid the processing gases from exiting through said interfaces, and force said process gases through the outlets 12 and then through the exhaust plenum

29. These seal means do not allow those process gases to enter the spaces 102, 103. The seal means are described in detail hereafter.

Considering an interface I between for instance the upper surface of the outer ring 10 and the cover plate 20, it is to be noted that the process gases, located in the first space 101 of the process chamber, form a gas flow $\Phi 1$ that has a pressure P1 slightly above the external atmospheric pressure, which results in that said gas flow $\Phi 1$ propagates in a first direction D1 and tends to exit from the process chamber 101 through said interface I. The seal means structures according to the invention first comprises the application of the counter-flow denoted $\Phi 2$ of inert gas that is introduced for instance by an inlet 39 into the second and third spaces 102, 103, around the outer ring 10, that has a pressure P2 slightly above the pressure P1, which results in that said gas flow $\Phi 2$ propagates in a second opposite direction D2 and tends to enter the process chamber 101 through said interface I. The counter-flow $\Phi 2$ of inert gas propagates through the interface I as a laminar flow with a parabolic speed profile. According to the parabolic flow propagation, the laminar flow speed V2 of the counter-flow is maximum in the middle of the small height of the interface I, while said laminar flow speed V2 is near zero along the so-called contact surfaces of the interface I. In that way, the first flow $\Phi 1$ that has an inferior pressure P1, is not allowed to exit the process chamber 101 through the interface I. It is known of those skilled in the art that the propagation of laminar gas flow is related to the height H of the interface I, and to the difference of pressure between the flow and the counter-flow and also that the speed V2 of the counter-flow $\Phi 2$ is function of this height H. Thus, a necessary speed V2 to make the counter-flow efficient implies that the difference of pressure between the flow and counter-flow or the squared height H^2 of the interface is not too small. It has been previously explained that the pressure difference $P2 - P1$ is not allowed to be great in order not to disturb the deposition process. Now, it is also important that the height H of the interface be not too large in order not to use a great amount of inert gas, which may also disturb the deposition process. As a matter of fact, it must be taken into account that the peripheral length of the processing chamber is important for accommodating several wafers, so that the amount of inert gas entering through the interface I may happen to be too great.

In order to provide a solution to all those problems, the seal means structures according to the invention comprises the formation, on at least one of the two opposite contact surfaces, of a set of substantially radial grooves, performed along a length at least equal to the radial dimension of the upper and lower surfaces of the outer ring, for the counter-flow $\Phi 2$ to enter the chamber 101 through said grooves. Now, the depth and the width of a groove, and the width between two adjacent grooves, are to be determined with respect to the length of the outer ring 10 in order to reach the aims of the invention. To that end, simulations are performed by calculation means of a standard kind according to known standard methods of calculation, which permit of determining those proper values. Instead

of grooves, roughened zones of the contact surfaces of the considered interface may be used. Thus, the functions of the outer ring is to both to constitute an isolating element and to comprise seal means structures allowing the entry of an appropriate counter-flow $\Phi 2$.

5 The counter-flow $\Phi 2$ propagates substantially through the grooves in the direction D2, i. e. from the space 102 towards the inside of the processing chamber 101. The gas flow $\Phi 1$, which cannot propagate through the grooves due to the value of the counter-flow $\Phi 2$, may however propagate between the grooves, through the still existing interface I. If the width between grooves is too important, said flow $\Phi 1$ may propagate through the whole length of the interface I from the chamber 101 until it reaches the space 102, which is
10 inconvenient. It why all the parameters of propagation of the counter-flow $\Phi 2$ and flow $\Phi 1$ are carefully calculated as above-described. When the appropriate ratio of the widths of the grooves and between the grooves is achieved, taking into account the length of the grooves, then, the gas flow $\Phi 1$ is no more allowed to exit the chamber 101 through the interface I, because, due to said appropriate ratio, the gas flow $\Phi 1$ cannot get throughout the whole
15 length of the grooves.

Referring to FIG.1, the counter-flow $\Phi 2$ is introduced by the inlet 39. It is shown that both the flow $\Phi 1$ and the counter-flow $\Phi 2$ are collected by the outlets 12, and then by the exhaust plenum 29. So, the gas flow $\Phi 1$ exits through the gas-collector 16, being not allowed to get through the interface I and the counter-flow $\Phi 2$ does not disturb
20 the process.

Preferably, in a reactor having a process chamber 101 and spaces 102, 103 surrounding the outer ring 10, the pressure difference $P2 - P1$ is about 10^{-5} to 10^3 mbars, which is considered a small difference of pressure; the height H of the interface I between closing surfaces is about 0 to 1 mm, preferably 0.05 to 0.1mm, which is considered small;
25 the width and the depth of the grooves are about 1mm and 0.3mm respectively; and the ratio giving the width between grooves is 5 times the width of the groove.

Referring to FIG.2 and FIG.3, in an other embodiment, the reactor comprises the base plate in two parts : a first ring-shaped base plate part 30A, which is disposed beneath the substrate holder 4A, and a second ring-shaped base plate part 30B, which
30 supports the first ring-shaped base plate part 30A, the gas-collector 16 and the outer ring 10. The second ring-shaped base plate part 30B is movable downwards with respect to the first ring-shaped base plate part 30A in order to create free access to the wafers above the outer ring and the gas-collector.

An aperture 40 is made in the peripheral wall 19C associated to a movable
35 window 19B, referred to as gate valve, in order to create an access to the wafers when the second ring-shaped base plate part 30B is in the lowest position shown FIG.3. For example, the arm of a robot may be used to manipulate the wafers.

The contact surfaces of the first ring-shaped base plate part 30A and the second ring-shaped base plate part 30B are provided with seal means structures as above-described.

Referring to FIG.4, in an other embodiment, the reactor comprises holes 55 and
5 58 provided in the outer ring 10 and the gas-collector 16 respectively, in order to introduce a tube 57. Through the tube 57, a light beam is guided for permitting of examining the wafers and for example of determining their rotation speed. The light beam may be introduced through the tube 57 for instance through a window 52, transparent to said light beam, and covering an aperture 53. The transparent window 52 may be bonded to the peripheral wall
10 19C at a part 51. Inside the tube 57, a transparent solid material, referred to as tube window 56, is inserted so as to permit the operation of the counter-flow $\Phi 2$ created by the difference of pressures between the second space and the first space each side of said tube window.

15

Claims:

A chemical vapor deposition reactor having a process chamber (101) accommodating a substrate holder (4a) for wafers (5), a first gas flow ($\Phi 1$) of reactive gases to process the wafers and a crown-shaped gas collector surrounding the substrate-holder, wherein said reactor further comprises :

a base plate (30) and a cover plate (20) disposed respectively beneath and above the substrate-holder,

an outer ring (10) surrounding the gas-collector (16) and touching both the base plate (30) and the cover-plate (20),

and a second flow ($\Phi 2$) of non-reactive gases propagating in spaces outside the process chamber referred to as first space (101), which first space is limited by the base and cover plates and the outer ring, and said second flow acting as a counter-flow ($\Phi 2$) for preventing the first reactive gas flow ($\Phi 1$) to exit from the process chamber but through the gas-collector (16).

2. A reactor as claimed in claim 1, wherein the outer ring (10) has a top and a low flat surfaces for touching respectively the cover and the base plates, and wherein said top and low surfaces are provided with grooves or roughened zones to permit the second gas flow of acting as a counter-flow ($\Phi 2$) through the interfaces between the outer ring and the cover and base plates.

3. A reactor as claimed in one of claims 1 or 2, wherein the substrate holder (4A) is supported by a rotating axis (4C), entering the process chamber (101) through a hole (32) in the base plate (30).

4. A reactor as claimed in Claim 3, wherein said axis is connected to the substrate holder (4A) by an annular part (4B) which is larger than the hole (32) in the base plate (30) for the rotating axis (4c), wherein said annular part (4B) connecting the substrate holder to the rotating axis is made of a material less porous than the substrate holder.

5. A reactor as claimed in claim 4, wherein the process temperatures are in the range of about 300 to 1500°C and wherein said annular part (4B) connecting the substrate holder (4A) to the rotating axis (4C) is made of a material further apt to withstand such high temperatures.

6. A reactor as claimed in one of claims 1 to 5, wherein the first space (101) is surrounded by second and third spaces limited by a substantially cylindrical peripheral wall (19C), a lower horizontal wall (19A) and a cover (28), wherein one wall comprises an inlet for the counter-flow ($\Phi 2$) and the cover comprises an inlet for the reactive gas flow ($\Phi 1$).

7. A reactor as claimed in one of claims 1 to 6, wherein the base plate comprises a first internal ring-shaped base plate part (30A) disposed beneath the substrate holder and a second peripheral ring-shaped base plate part (30B) supporting the first ring-shaped base plate part (30A), the gas-collector (16) and the outer ring (10), the second ring-shaped base

plate part (30B) being movable with respect to the first ring-shaped base plate part (30A) in a downward direction in order to create free access to the wafers for a manipulation operation of the wafers.

5 8. A reactor as claimed in Claim 7, wherein the peripheral wall (19A) comprises a gate valve to create access to the wafers.

9. A reactor as claimed in one of Claims 7 or 8, wherein the contact surfaces of the first and second ring-shaped base plate parts (30A, 30B) are provided with grooves or roughened zones to permit the second gas flow ($\Phi 2$) of acting as a counter-flow through the interface between said contact surfaces.

10 10. A reactor as claimed in one of claims 1 to 9, wherein a tube (57) is inserted through holes (55, 58) respectively made through the outer ring (10) and the gas-collector (16) in order to introduce a light beam for examining the wafers on the substrate-holder.

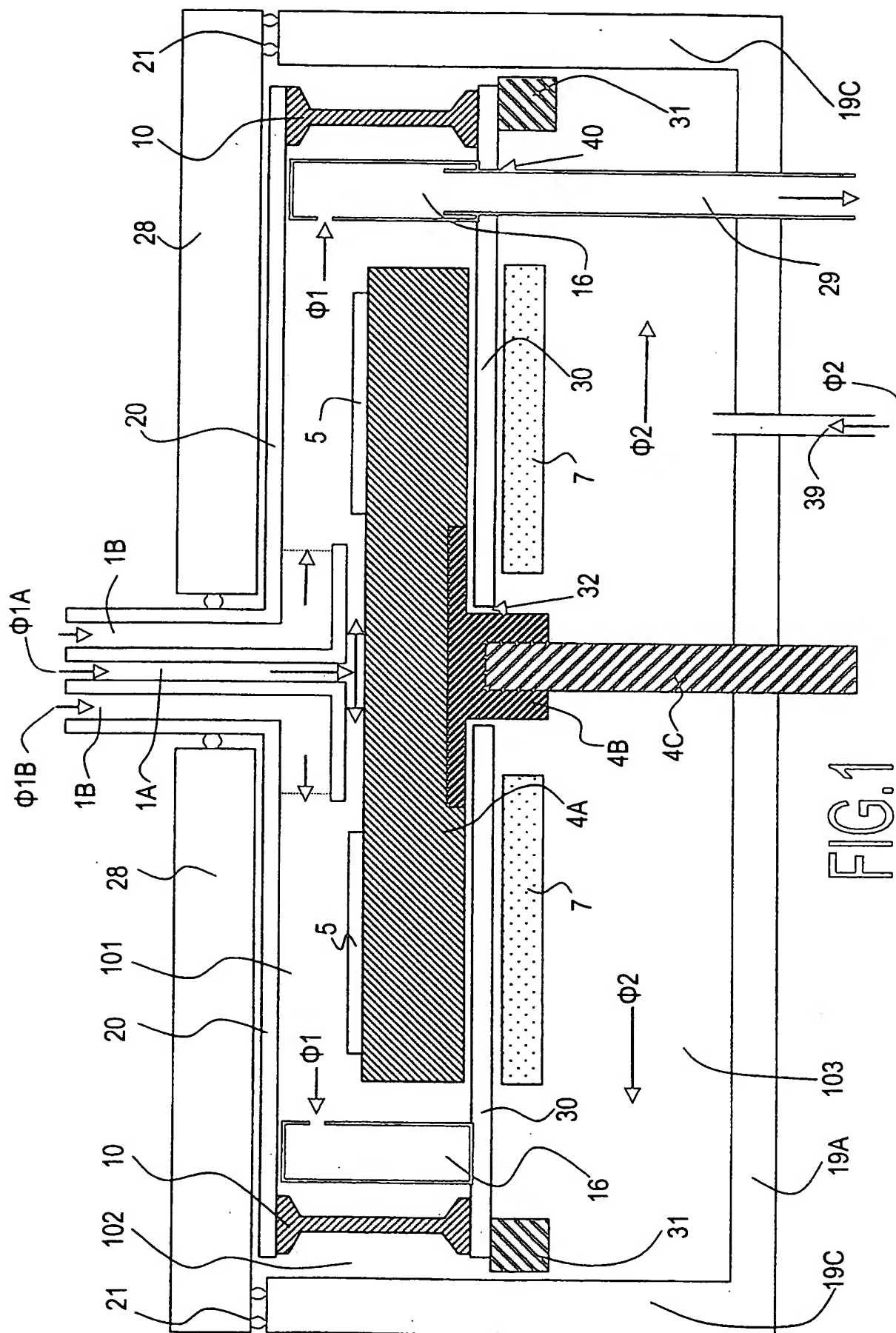
11. A reactor as claimed in Claim 10, wherein a piece of solid material (56), denoted tube window, transparent to the light beam, is inserted inside the tube (57) in such
15 a manner that the contact surfaces of the tube and tube window permit the second flow ($\Phi 2$) to act as a counter-flow for preventing the first reactive gas flow from exiting the process chamber (101).

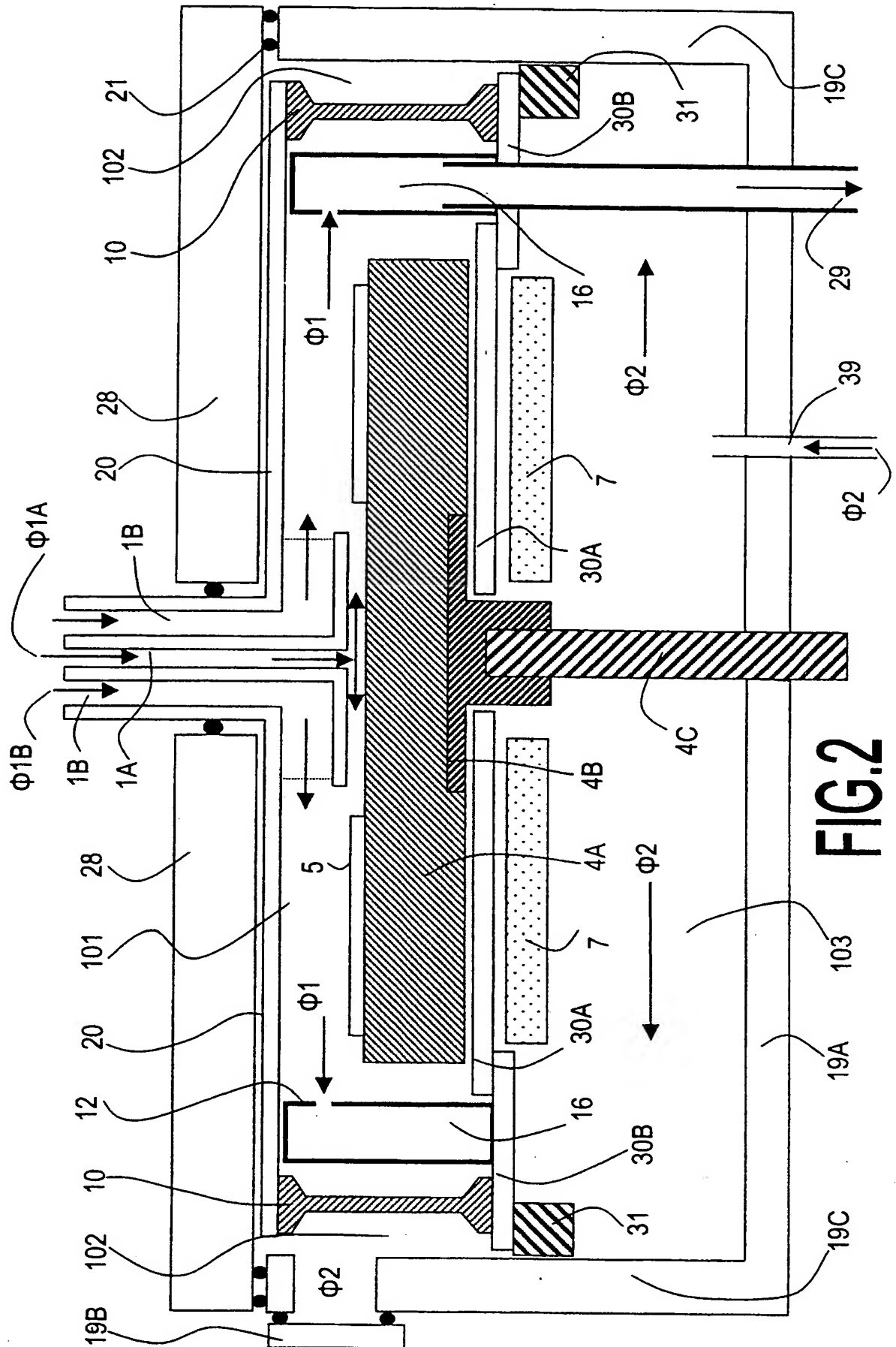
12. A reactor as claimed in Claim 11, wherein the cylindrical peripheral wall (19C) has a window (52) transparent to the light beam to introduce said light beam through the
20 tube (57).

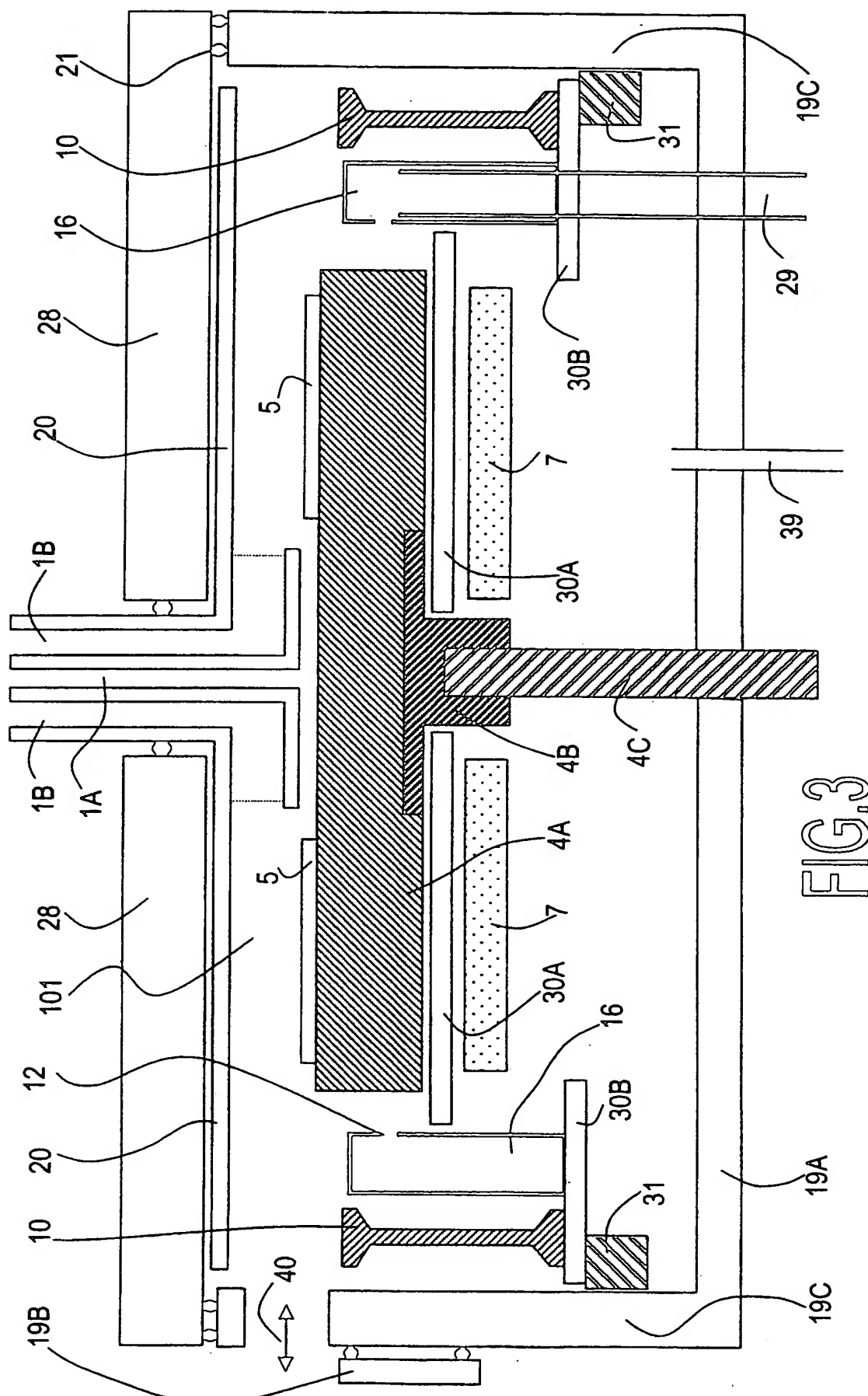
13. A reactor as claimed in one of claims 1 to 12, wherein heating means (7) are provided beneath the base plate for heating the substrate holder.

14. A reactor as claimed in one of claims 1 to 13, wherein the gas collector is constituted of an annular top part (15), an annular low part (14), said top and low parts
25 being connected by lateral flanges (17, 18) and hold together by screws (13), and wherein the flange facing the substrate holder comprises outlets (12) for the first reactive gas flow.

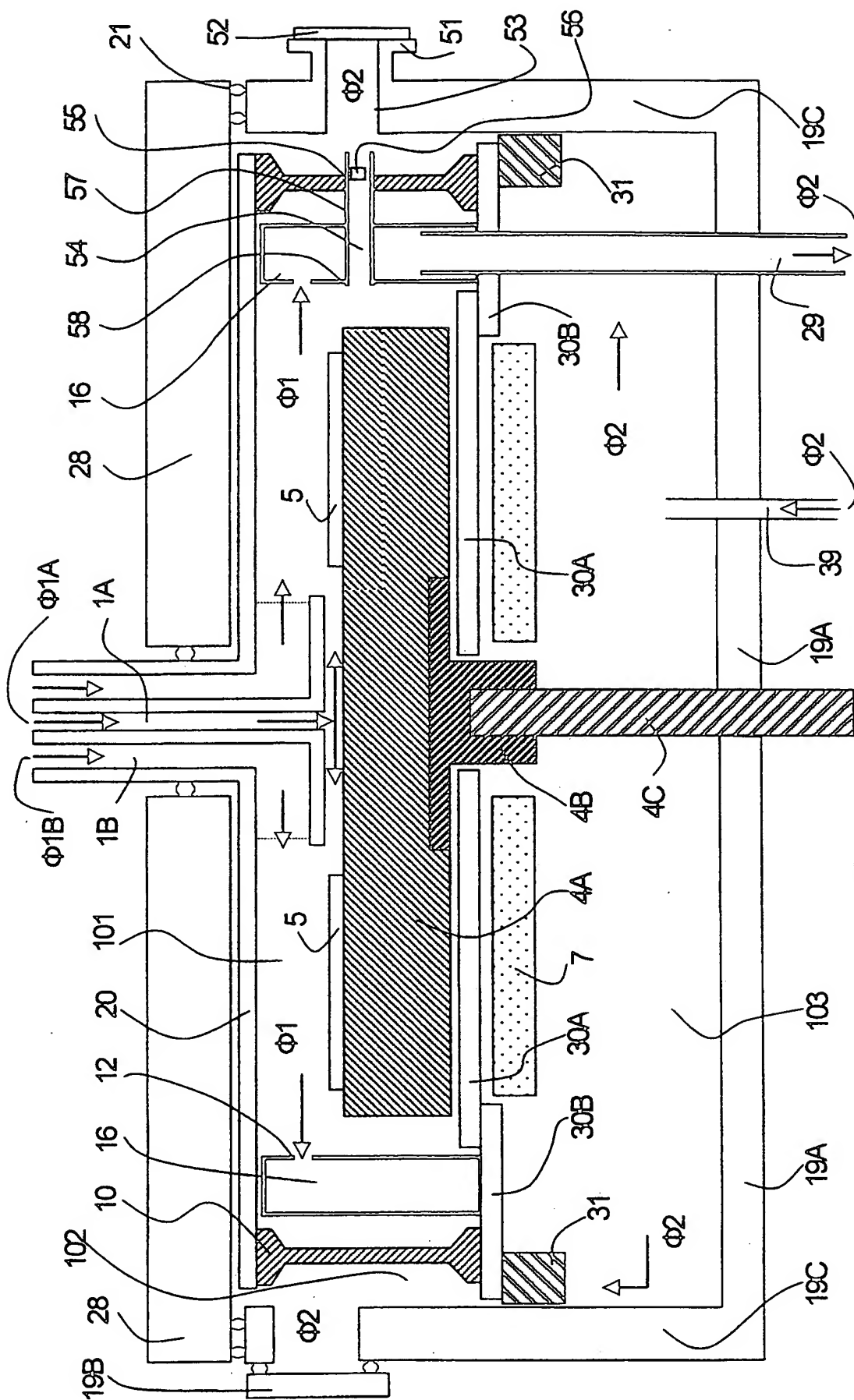
15. A reactor as claimed in one of claims 1 to 14, wherein a gas plenum (29) for collecting the gases from the gas-collector comprises a tube (29) sliding in holes (40) of the gas-collector and base plate, and said tube (29) having walls (29A) provided with ridges
30 (29B) creating a space between the tube (29) and the edges (41) of the holes (40) to permit the second gas flow ($\Phi 2$) of acting as a counter-flow.







36



5/5

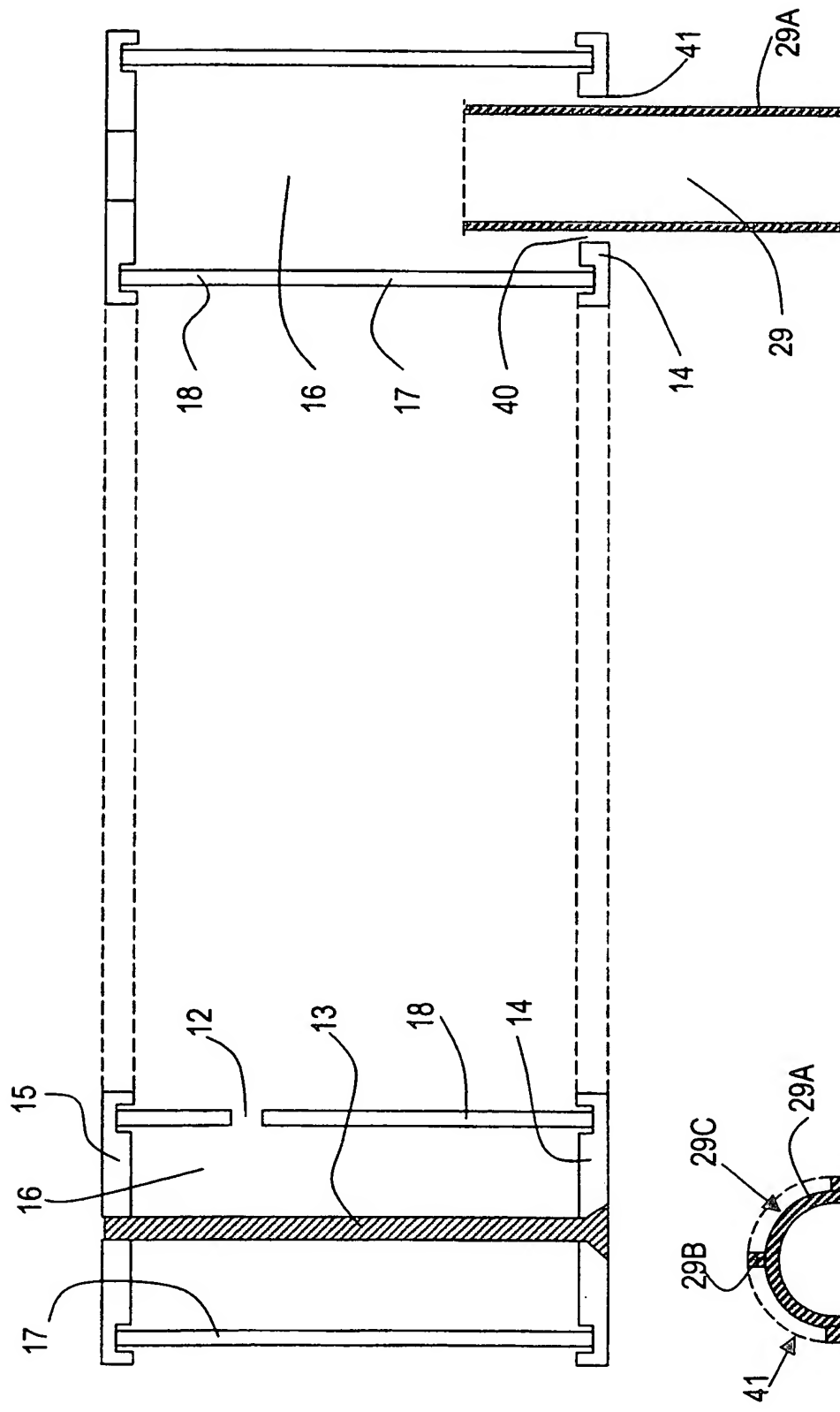
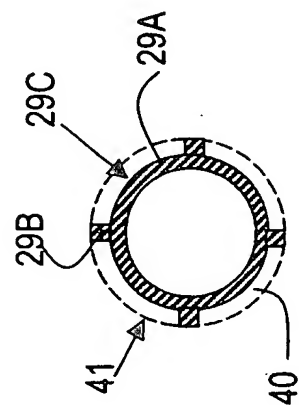


FIG. 5A

FIG. 5B



**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☐ BLACK BORDERS
- ☐ IMAGE CUT OFF AT TOP, BOTTOM OR SIDES
- ☒ FADED TEXT OR DRAWING
- ☒ BLURRED OR ILLEGIBLE TEXT OR DRAWING
- ☐ SKEWED/SLANTED IMAGES
- ☐ COLOR OR BLACK AND WHITE PHOTOGRAPHS
- ☐ GRAY SCALE DOCUMENTS
- ☒ LINES OR MARKS ON ORIGINAL DOCUMENT
- ☒ REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY
- ☐ OTHER: _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.